

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: James L. Piller et al.

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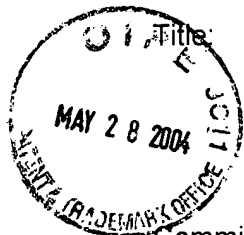
Examiner: E W Thomas

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Group Art Unit: 2831

Title: Improving ESR of Solid  
Electrolytic Capacitors Using  
Conductive Polymer Cathodes

Confirmation No.: 2720



DECLARATION UNDER 37 CFR §1.132

Commissioner for Patents  
Mail Stop Non-fee Amendment  
P.O. Box 1450  
Alexandria VA 22313-1450

Dear Sir:

I Erik Reed, do hereby declare:

1. That I have been employed by Kemet Electronics since 1976;
2. That I hold a Bachelor's Degree in Broadcast Engineering from Bob Jones University, awarded in 1976;
3. That I hold a Bachelor's Degree, Master's Degree and Doctoral Degree in Electrical Engineering, awarded in 1985, 1988 and 1990 respectively;
4. That I was a Research Associate at Lawrence Livermore National Laboratory in 1987 and 1988;
5. That I was a visiting Assistant Professor of Electrical Engineering at Clemson University in 1990 and 1991;
6. That under my direction, the experiments described *infra* were performed by George Haddox, a staff engineer with 26 years experience in the design and fabrication of testing devices for electronic components;
7. The objective of these experiments was to determine whether adhesion in the carbon layer and at the interface of a carbon coating and an underlying conductive polymer cathode layer could be improved by infusing the carbon layer with a conductive polymer;

8. A test stand was made using a Chatillon LTCM-6 motorized test stand ([www.chatillon.com](http://www.chatillon.com)) and a Chatillon DFGS10 integrated loadcell digital force gauge, both of which were attached to a PC using the original equipment RS 232 serial communication ports. A board was used to mount each test sample. Adjacent to the mounting location, a thermocouple was secured to measure temperature at the time of the test. A small hot air blower was used to adjust test temperature.

Samples were mounted with one face attached to the board using an epoxy adhesive. An eyelet having a diameter of 0.075 inch was soldered to a 24 ga. wire and attached to each test sample by epoxy adhesive. The wire was pulled using the motorized test stand;

9. Test Sequence A:

A control group of capacitors was prepared as follows:

- a) a Ta anode coupon was etched to form an oxide coating;
- b) a conductive polymer of 3,4-polyethylenedioxythiophene was coated on the coupon via a sequential oxidizer/monomer dip process. After polymerization the coupons were washed and the cycle repeated to build up sufficient conductive polymer on all internal and external dielectric surfaces;
- c) the product of step b.) above was dipped into a colloidal graphitized carbon suspension;
- d) the product of step c.) was dipped into an Ag paint and dried;

Test samples were prepared by following steps a through c above, then dipped into a oxidizer solution, allowed to sit at room temperature for approximately 1 hour to allow the solvent to dry, dipped in a monomer solution and allowed to sit at room temperature so that the oxidizer could polymerize the monomer (sequential oxidizer/monomer dip process). Following the polymerization reaction the coupon was washed to remove reaction by-products. The modified coupon was then treated according to step d.) above;

RESULTS: TABLE 1

Temperature	Test Sample		Control	
	Average Separation Force (gm)	Break Point	Average Separation Force (gm)	Break Point
RT	300	Carbon/silver interface	300	Carbon/silver interface
80° C	20	Carbon/silver interface	20	Carbon/silver interface
100° C	5	Carbon/silver interface	5	Carbon/silver interface

## 10. Test Sequence B

In order to measure the adhesive strength of the polymer/carbon layers and interface the eyelet was attached to the portion of face of control and test samples where the silver had been removed from the face of the anode by test sequence A. The bottom face of the anode with silver intact remained adhered to the test board. All pulls were conducted at 100°C.

Sample	Test Sample		Control	
	Separation Force (gm)	Break Point	Separation Force (gm)	Break Point
1	100	Carbon/silver interface on bottom side of sample	25	In the carbon
2	115	Carbon/silver interface on bottom side of sample	55	In the carbon
3	115	Carbon/silver interface on bottom side of sample	75	In the carbon
4	125	Carbon/silver interface on bottom side of sample	120	In the carbon

## 11. Test Sequence C

In order to measure the adhesive strength of the polymer/carbon layers and interface of the test samples (which in test sequence B separated between the carbon and silver on the bottom side of the sample rather than in the polymer/carbon layers) the test samples from test sequence B were reattached to the test board where the silver had been removed from the bottom face of the anode. All pulls were conducted at 100°C.

Sample	Test Sample	
	Separation Force (gm)	Break Point
1	150	Polymer layer and polymer/carbon interface
2	200	Polymer layer and polymer/carbon interface
3	415	Polymer layer and polymer/carbon interface
4	525	Polymer layer and polymer/carbon interface

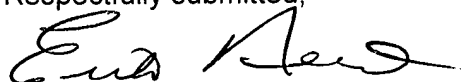
## 12. Conclusions

- The silver-carbon interface is very weak, especially above 100°C.
- The post carbon polymerization cycle does not improve silver bonding to carbon.
- The strength of the polymer/carbon layers, including the interface, is greatly enhanced by the addition of a post carbon polymerization cycle.

In each case, the separation occurred on the side where the Ag layer was attached to the test fixture and not at the exposed carbon side.

Declarant affirms that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true and further acknowledge that willful false statements and the like are punishable by fine or imprisonment, or both (18 USC 1001) and may jeopardize the validity of the application or any patent issuing therefrom.

Respectfully submitted,



Erik Reed, Ph.D.